

Organic
Chemical

Moisture of Halogenated Hydrocarbons

Volumetric titration (Direct Method) by
Karl Fischer Moisture Titrator

Standard	JIS	K 0113
	ASTM	E 203
	ISO	760

1. Abstract

Moisture titration using Karl Fischer reagent is popularly practiced water determination worldwide as the most reliable method. The procedure is adopted in many official standards as test method specified in ISO, ASTM, DIN, BS and JIS.

The test conducted this time is an example of volumetric moisture titration according to JIS K 0113-2005 for measurement of water content in Halogenated hydrocarbon, which dissolves with ease in ML or MI extracting medium.

But for those Halogenated hydrocarbons with long chain which are hard to dissolve in methanol, solvent CM (for oil) containing chloroform is used in titration.

Tested chemicals this time are as follows:

Chloroform / Carbon tetrachloride / Dichloromethane / 1,2 dichloroethane /
Isopropyl bromide / Ethyl bromide / n-butyl bromide / Benzene bromide /
Ethane iodide / n-butyl chloride / o-dichlorobenzene /
1,1,2,2-tetrachloroethane / 1,1,2-trichloroethane / Trichloroethylene /
1,1,1-trichloroethane / Ethylene tetrachloride

2. Reference

- 1) JIS K 0113-2005: Standard Test Method by Potentiometric, Amperometric, Coulometric and Karl Fischer Titration
- 2) ASTM E 203-16 Standard Test Method for Water Using Volumetric Karl Fischer Titration
- 3) ISO 760:1978 Determination of Water-Karl Fischer method (General method)
- 4) Hydranal manual published by Riedel de Haen

3. Cautions in measurement

- 1) In order to refrain from the effect of ambient humidity, the test must be conducted in a well air-conditioned room.
- 2) Handle with care when you work on chemicals.
- 3) For sample injection, change the side stopper of flask to syringe type.
- 4) For those materials of low water content like Halogenated hydrocarbon, use Karl Fischer reagent of low factor.
- 5) Obtain the factor of Karl Fischer reagent using the solvent in advance.

4. Post-measurement care

After the reagent is drained out and the electrode is cleaned, keep the titration flask filled with extracting solvent.

5. Test equipment

Main unit : Karl Fischer moisture titration volumetric system

Electrode : Twin platinum electrode for KF titration

6. Reagent

Titrant : Hydranal Composit 2 (Riedel de Haen)

Solvent : Extracting medium MI (for general purpose) (Hayashi Chemicals)

7. Measurement procedure

-Pretreatment-

- 1) Prepare approximately 30mL extracting medium in the titration cell.
- 2) Dehydrate the measuring cell by performing pre-titration in advance.

-Measurement-

- 1) Prepare approximately 10mL sample with a syringe.
- 2) Weigh the syringe on an electronic balance of reading to the nearest 0.1mg.
- 3) Transfer the sample in syringe to titration cell to dissolve.
- 4) Press Start key of titration unit.
- 5) Weigh the syringe of the above 3).
- 6) Enter the weight of item 2) for Wt1, and 5) for Wt2.
- 7) Obtain water content from titration volume, of which EP is detected automatically.

8. Formula

$$\text{Moisture (\%)} = ((\text{Data} \times F - \text{Blank}) / (\text{Wt1} - \text{Wt2})) \times 0.1$$

Data : Titration volume (mL)

F : Factor of titrant (mg H₂O / mL)

Blank : Blank level (mg)

Wt1 : Sample + Syringe (g)

Wt2 : Empty syringe (g)

9. Example of measurement

-Titration parameter-

MKV-710M/S,MKA-610	MKA-520	MKS-500
Method No. 1 [Titration] Titr.mode Normal t(stir) 0 s t(wait) 10 s t(max) 0 s t(interval) 0 s Max.volume 10 mL Titr.bur.No. 1 Dose mode Off [Control] End time 30 s Final vol. 0.01 mL Titr.speed 3 Detect.mode 1 Drift titr. On Start mode Manual End level 75 mV Samp.time 5 s Stir.speed 4	[Titration] Method 1 Titr Mode Normal Titr Buret No. 1 End Time 30 s Final Vol. 0.01 mL Titr.Speed 3 Detector Mode 1 t(stir) 0 s t(wait) 10 s t(max) 0 s Drift Titr On Start Manual Max.Volume 10 mL Dose mode Off Oven Off	[Titration] Method Direct Titr.Speed 3 End Time 30 s Final Vol. 0.01 mL Detector Mode Normal t(stir) 0 s t(max) 0 s Drift Titr. On Max.Volume 10 mL

-Calculation parameter-

MKV-710M/S,MKA-610	MKA-520	MKS-500
[Calculation] Calc.type Sample Blank No. 1 Calc.No. 2 Unit % Decimal 3 Fraction Round (Half adjust) Drift comp. Off Evaluation Off	[Calculation] Calc. 2 Unit % Weight Variable	[Calculation] g->%

—Measurement results—

Sample name	Water content	
	mg	%
Chloroform	0.69	0.005
Carbon tetrachloride	0.80	0.005
Dichloromethane	0.99	0.007
1,2 dichloroethane	0.54	0.004
Isopropyl bromide	2.51	0.019
Ethyl bromide	5.81	0.040
n-butyl bromide	1.28	0.010
Benzene bromide	2.99	0.020
Ethane iodide	2.13	0.011

Sample name	Water content	
	mg	%
Iodized methane	0.46	0.002
n-butyl chloride	0.53	0.006
o-dichlorobenzene	0.66	0.005
1,1,2,2-tetrachloroethane	0.58	0.005
1,1,2-trichloroethane	1.27	0.008
Trichloroethylene	0.59	0.004
1,1,1-trichloroethane	0.27	0.002
Ethylene tetrachloride	0.33	0.002

Solvent MI is used for extracting medium.

10. Summary

Halogenated hydrocarbons are compounds with Halogens (Chlorine · Bromine · Fluorine · Iodine, etc.) instead of Hydrogen for Hydrocarbon.

The test sample in this application dissolves in solvent MI, which makes moisture titration performed with ease.

Stable measurement of water content is assured by Karl Fischer moisture titration.